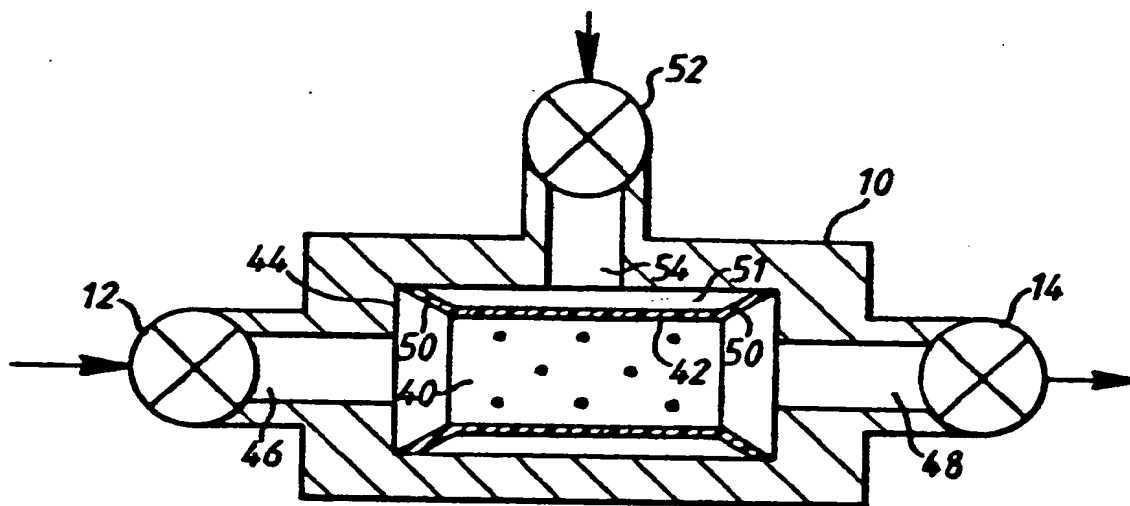




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

| | | |
|---|-----------|---|
| (51) International Patent Classification 4 : G01N 1/28, 15/08, 33/24 | A1 | (11) International Publication Number: WO 87/ 04790 (43) International Publication Date: 13 August 1987 (13.08.87) |
| (21) International Application Number: PCT/GB87/00084 (22) International Filing Date: 5 February 1987 (05.02.87) (31) Priority Application Number: 8603157 (32) Priority Date: 8 February 1986 (08.02.86) (33) Priority Country: GB (71) Applicants (for all designated States except US): EDINBURGH PETROLEUM DEVELOPMENT SERVICES LIMITED [GB/GB]; Heriot-Watt University Research Park, Riccarton, Edinburgh (GB). HERIOT-WATT UNIVERSITY [GB/GB]; Riccarton, Edinburgh (GB). (72) Inventors; and (75) Inventors/Applicants (for US only) : McLEOD, Charles [GB/GB]; 13 Humbie Road, Kirkliston, Edinburgh (GB). McPHEE, Colin, Alexander [GB/GB]; 99 Curriehill Castle Drive, Balerno, Edinburgh (GB). | | (74) Agent: FITZPATRICKS; 4 West Regent Street, Glasgow G2 1RS (GB). (81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (European patent), LU (European patent), NL (European patent), NO, SE (European patent), US. Published <i>With international search report.</i> |

(54) Title: METHOD AND APPARATUS FOR PREPARATION OF ROCK CORE SAMPLES

**(57) Abstract**

A rock core sample (40) is enclosed within a chamber (44) having inlet and outlet ports (46, 48). A cleaning solvent is flushed through the chamber (44) to clean the sample (40) which is then dried by flushing a transition fluid (such as liquid carbon dioxide) through the chamber (44), closing the inlet and outlet valves (12, 14), raising the temperature and pressure of the fluid within the chamber (44) beyond the critical point thereof, and reducing the pressure of the fluid below the critical pressure thereof, by opening the outlet valve (14), such that the transition fluid changes smoothly from liquid to gas without going through a liquid-vapour phase, thereby reducing the risk of damage to the structure of the sample.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

| | | | | | |
|----|------------------------------|----|--|----|--------------------------|
| AT | Austria | FR | France | ML | Mali |
| AU | Australia | GA | Gabon | MR | Mauritania |
| BB | Barbados | GB | United Kingdom | MW | Malawi |
| BE | Belgium | HU | Hungary | NL | Netherlands |
| BG | Bulgaria | IT | Italy | NO | Norway |
| BJ | Benin | JP | Japan | RO | Romania |
| BR | Brazil | KP | Democratic People's Republic of Korea | SD | Sudan |
| CF | Central African Republic | KR | Republic of Korea | SE | Sweden |
| CG | Congo | LI | Liechtenstein | SN | Senegal |
| CH | Switzerland | LK | Sri Lanka | SU | Soviet Union |
| CM | Cameroon | LU | Luxembourg | TD | Chad |
| DE | Germany, Federal Republic of | MC | Monaco | TG | Togo |
| DK | Denmark | MG | Madagascar | US | United States of America |
| FI | Finland | | | | |

- 1 -

METHOD AND APPARATUS FOR PREPARATION OF ROCK CORE SAMPLESDescription

The present invention is concerned with the preparation of rock core samples prior to analysis, such as porosity and permeability measurements or scanning electron microscope analysis, and is particularly intended for use with reservoir samples obtained in connection with petroleum exploration.

The preparation of rock samples for laboratory analysis involves cleaning, to remove brine and crude oil or the like from the sample, and drying, to remove the cleaning fluids. It is important that the original properties of the sample are not altered by the preparation process since this may invalidate the results of any subsequent analysis.

A common problem with preparation techniques currently employed is that clays which may be contained within the sample are subject to both chemical and physical damage, which can substantially alter the porosity and permeability of the sample. Chemical damage can be avoided by careful selection of cleaning solvents; however the normal drying process, heating the core in an oven to evaporate the solvents, may physically damage the clays in the sample by the formation of gas/liquid interfaces and by the removal of their natural water content by overheating. Furthermore, known techniques are imprecise, and generally very slow and thus expensive in terms of time and resources.

It is an object of the present invention to obviate or mitigate the aforesaid disadvantages.

In accordance with one aspect of the present invention, a method of preparing a rock core sample for analysis comprises the steps of:

confining the sample in a chamber having an inlet port and an outlet port;

flushing at least one cleaning solvent through said chamber via said inlet and outlet ports;

passing a transition fluid, in its liquid state, through said chamber via said inlet and outlet ports;

5 increasing the temperature and pressure of said transition fluid within said chamber beyond the critical point thereof; and

reducing the pressure of said transition fluid within said chamber below the critical pressure thereof.

10 The temperature and pressure are preferably increased beyond the critical point by closing the inlet and outlet ports and applying heat to the closed chamber.

Preferably, said transition fluid is miscible with the cleaning solvent or solvents used.

15 Preferably also, said transition fluid is carbon dioxide.

Preferably also, said cleaning solvent is inspected at the outlet port of said chamber and flushing is continued until the effluent solvent is clean.

20 In a particularly preferred embodiment, the rock core sample is closely confined within said chamber such that substantially all of the cleaning solvent passes through said sample, and wherein the liquid permeability of the sample is monitored during cleaning by measuring
25 the pressure differential across the sample.

Preferably also, said solvent is pumped through said chamber against a controllable, variable back-pressure.

In accordance with a second aspect of the present invention, apparatus for the preparation of rock core
30 samples for analysis comprises;

a sample holder defining a closed chamber having an inlet port and an outlet port;

valve means associated with said inlet and outlet ports;

35 means for flushing at least one cleaning solvent

through said chamber via said inlet and outlet ports;

means for passing a transition fluid through said chamber via said inlet and outlet ports; and

5 means for controlling the temperature and pressure of said transition fluid within said chamber.

Preferably, the apparatus includes flow directing means located within said chamber comprising a sleeve of flexible material into which the sample fits closely, the longitudinal ends of said sleeve being adapted to
10 engage the interior walls of said chamber such that fluid entering the inlet port is directed into the interior of said sleeve and hence through the sample.

Preferably also, means are also included for applying a confining pressure to the exterior of said
15 sleeve.

In a particular embodiment for the treatment of a right cylindrical sample, said sleeve is generally tubular, the longitudinal ends thereof being flared outwardly to engage said interior walls of said chamber.
20

Preferably also, means are also included for measuring the pressure differential across said sample.

Alternatively, the sample holder is adapted to receive a variety of interchangeable chambers having different interior shapes and volumes.

25 Preferably also, said apparatus includes a plurality of solvent reservoirs and distributor means for selectively flushing solvent from one of said reservoirs through said sample holder.

Preferably also, said apparatus includes means
30 for inspecting the effluent cleaning solvent drained from the outlet of the sample holder.

Preferably also, means are included for applying a controllable, variable back pressure to said sample holder.

35 Embodiments of the invention will now be described,

by way of example only, with reference to the accompanying drawings, in which:-

Fig. 1 is a schematic representation of apparatus for cleaning a rock core sample in accordance with the invention;

Fig. 2 is a schematic representation of apparatus for drying a rock core sample after cleaning; and

Fig. 3 is a schematic drawing, partly in section, of a preferred embodiment of a sample holder for use with the apparatus of Figs. 1 and 2.

Referring now to Fig. 1 of the drawings, apparatus for cleaning a rock core sample includes a sample holder 10 having a sample chamber (not shown) therein which communicates via inlet and outlet ports (not shown) with inlet and outlet valves 12 and 14. The inlet valve 12 is connected, via a pump 16 and distributor 18 to a plurality of cleaning solvent reservoirs 20. Filters 22 are provided between the reservoirs 20 and the distributor 18. The reservoirs may be pressurised with nitrogen via line 24 to suppress fumes and are also provided with closeable vents 26 to atmosphere.

In use, a sample is placed in the chamber of the sample holder 10, one of the solvent reservoirs 20 is selected by means of the distributor 18, the inlet and outlet valves 12 and 14 are opened, the pump 16 started and the chosen cleaning solvent is pumped through the sample holder 10 to clean the sample. Suitably a device (not shown) is included in the outlet line for providing a selected back pressure, which can be adjusted in accordance with the permeability of the sample under measurement.

The effluent solvent may be collected temporarily in an inspection sump 28 where its cleanliness may be evaluated visually or by automated means such as a UV spectrophotometer (not shown). The sample is judged

- 5 -

to be clean when the solvent is unchanged after passing through the sample holder 10.

Any one of a variety of solvents contained in the reservoirs 20 may be selected or a chosen sequence of solvent may be employed as required. Each solvent in a sequence should be miscible with the preceding solvent, and the final solvent miscible with the transition fluid used in the drying operation to be described.

Once the sample is clean the pump may be deactivated, the valves 12 and 14 closed, and the sample holder 10 can then be removed from the cleaning apparatus for drying as described below.

Referring now to Fig. 2 the sample holder 10 containing the cleaned sample and residual solvent is placed in a closed canister 30 and the inlet valve 12 is connected to a source of transition fluid, such as liquid carbon dioxide, via line 32 and valve 33 and the outlet valve to a drain via line 34 and valve 35. The transition fluid is chosen to be miscible with the cleaning solvent.

The temperature of the sample holder is controlled by carbon dioxide let into the canister via line 36 and valve 38, which is preferably a solenoid valve controlled by a microprocessor.

The sample is dried as follows: the inlet and outlet valves 12 and 14, and valves 33 and 35 are opened and the transition fluid, in its liquid state, is passed through the sample holder 10 to displace the residual solvent. Thereafter the inlet and outlet valves 12, 14 are closed and the temperature of the closed system is raised thus causing the pressure of the transition fluid within the sample holder also to increase until temperature and pressure are beyond its critical point (i.e. 31°C and 1071 psi for carbon dioxide). The outlet valve 14 is then reopened, allowing the pressure

to fall at a controlled rate below the critical pressure so that the transition fluid changes smoothly from liquid to gas without going through a liquid-vapour phase. In this way the sample is dried without any gas-liquid
5 interfaces being formed which might damage the sample and no high temperatures are involved.

A preferred form of the sample holder 10 is shown schematically in Fig. 3. This embodiment is suitable for use with right-cylindrical cores and improves the
10 efficiency of the cleaning and drying processes.

In this case, the cylindrical core 40 is placed in a close-fitting, substantially cylindrical, flexible sleeve 42 which is in turn placed in a cylindrical chamber 44 in the sample holder 10 which, again, is
15 provided with inlet and outlet ports 46 and 48 and valves 12 and 14.

The longitudinal ends 50 of the sleeve 42 are flared outwards so that they engage the walls of the chamber 44. Thus, any treatment fluid, whether cleaning
20 solvent or transition fluid, entering the chamber from the inlet port 46 is directed through the core 40. A confining pressure is applied to the exterior of the sleeve 42 - by means of a pressurising fluid, such as nitrogen, pumped into the annular space 51 around the
25 sleeve 42 via a second inlet valve 52 and port 54 - so as to prevent distortion of the sleeve 42 and by-passing of the core 40 by the treatment fluid. The confining pressure is typically twice the pressure of the treatment fluid.

This arrangement also allows the liquid permeability of the core 40 to be monitored during cleaning, by measuring the pressure differential across the sample. Preferably, sensors (not shown) are provided to measure solvent line pressure, temperature and flow rate
35 electronically via a dedicated microprocessor control to

provide data for measuring permeability and to assist in identifying and quantifying core damage.

5 Samples for use in scanning electron microscope analysis are generally small fragments of irregular shape and hence it is impractical to tailor the shape of the chamber to fit them closely. In this case, a series of interchangeable chambers of varying internal shape and size may be provided and the most suitable of these chosen in accordance with the dimensions of the particular
10 sample. It is also preferred in this case to perform both cleaning and drying in a combined operation in one location, which reduces the potential of damage to the sample.

15 In addition to flushing the solvent through the chamber, it may be useful in some cases to raise and lower the pressure of the solvent, e.g. by pumping the solvent in against a high back pressure which is subsequently released. This assists penetration of the solvent into the pores.

20 The present invention, then, provides a method and apparatus for the preparation of rock core samples wherein the sample is cleaned and dried in the same chamber and wherein the speed and efficiency of the process is greatly improved; the invention also
25 facilitates permeability measurement during cleaning.

Claims

1. A method of preparing a rock core sample (40)
for analysis comprising the steps of:
 confining the sample in a chamber (44) having
5 and inlet port (46) and an outlet port (48);
 flushing at least one cleaning solvent through
said chamber (44) via said inlet and outlet ports (46, 48);
 passing a transition fluid, in its liquid state,
through said chamber (44) via said inlet and outlet ports
10 (46, 48);
 increasing the temperature and pressure of said
transition fluid within said chamber (44) beyond the
critical point thereof; and
 reducing the pressure of said transition fluid
15 within said chamber (44) below the critical pressure
thereof.
2. A method as claimed in claim 1 wherein the
temperature and pressure of said transition fluid is
raised beyond the critical point by closing said inlet
20 and outlet ports (46, 48) and applying heat to the closed
chamber (44).
3. A method as claimed in claim 1 or claim 2
wherein said transition fluid is miscible with said
cleaning solvent.
- 25 4. A method as claimed in any of claims 1, 2 or 3
wherein said transition fluid is carbon dioxide.
5. A method as claimed in any preceding claim
wherein said cleaning solvent is inspected after leaving
said outlet port (48) and flushing is continued until the
30 effluent solvent is clean.
6. A method as claimed in any preceding claim
wherein the rock core sample (40) is closely confined
within said chamber (44) such that substantially all of
the cleaning solvent passes through said sample, and
35 wherein the liquid permeability of the sample (40) is

monitored during cleaning by measuring the pressure differential across the sample (40).

7. A method as claimed in any preceding claim wherein said solvent is pumped through said chamber (44)

5 against a controllable, variable back-pressure.

8. Apparatus for the preparation of a rock core sample (40) for analysis comprising:

a sample holder (10) defining a closed chamber (44) having an inlet port (46) and an outlet port (48);

10 valve means (12, 14) associated with said inlet and outlet ports (46, 48);

means (16 to 24) for flushing at least one cleaning solvent through said chamber (40) via said inlet and outlet ports (46, 48);

15 means (32 to 35) for passing a transition fluid through said chamber (44) via said inlet and outlet ports (46, 48); and

means (30, 36, 38) for controlling the temperature and pressure of said transition fluid within said
20 chamber (44).

9. Apparatus as claimed in claim 8 including flow directing means located within said chamber (44) comprising a sleeve (42) of flexible material into which the sample (40) fits closely, the longitudinal ends (50)
25 of said sleeve being adapted to engage the interior walls of said chamber (44) such that fluid entering the inlet port (46) is directed into the interior of said sleeve (42) and hence through the sample (40).

10. Apparatus as claimed in claim 9 further including
30 means (52, 54) for applying a confining pressure to the exterior of said sleeve (42).

11. Apparatus as claimed in claim 9 or claim 10 for the treatment of a right-cylindrical sample (40) wherein said sleeve (42) is generally tubular, the
35 longitudinal ends (50) thereof being flared outwardly

to engage said interior walls of said chamber (44).

12. Apparatus as claimed in any of claims 9, 10 or 11, further including means for measuring the pressure differential across said sample (40).

5 13. Apparatus as claimed in claim 8 wherein said sample holder 10 is adapted to receive a variety of interchangeable chambers having different interior shapes and volumes.

10 14. Apparatus as claimed in any of claims 8 to 13, further including a plurality of solvent reservoirs (20) and distributor means (18) for selectively flushing solvent from one of said reservoirs (20) through said sample holder (10).

15 15. Apparatus as claimed in any of claims 8 to 14, further including means (28) for inspecting the effluent cleaning solvent drained from the outlet (48) of said sample holder (10).

20 16. Apparatus as claimed in any of claims 8 to 15 further including means for applying a controllable, variable back pressure to said sample holder (10).

1/1

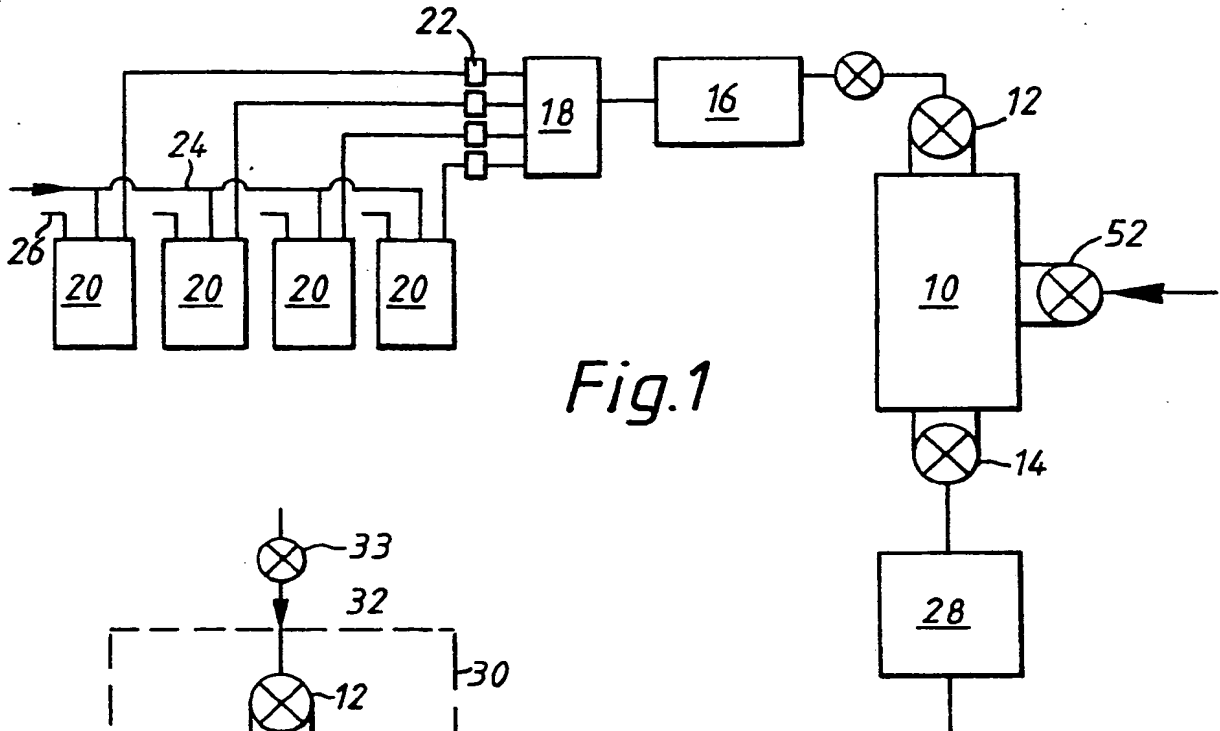


Fig. 1

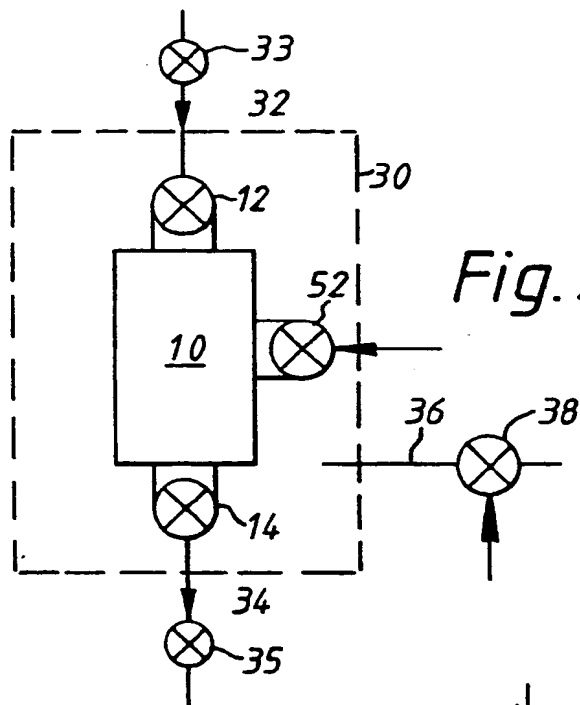


Fig. 2

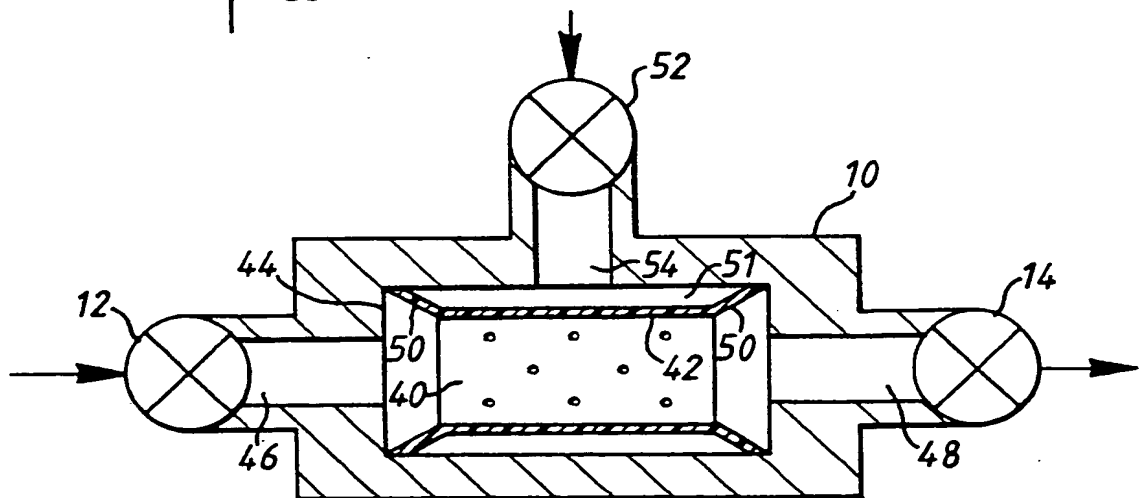
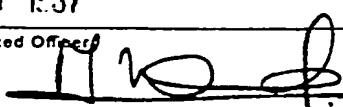


Fig. 3

SUBSTITUTE SHEET

INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 87/00084

| | | |
|--|--|-------------------------------------|
| I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) * | | |
| According to International Patent Classification (IPC) or to both National Classification and IPC | | |
| IPC ⁴ : G 01 N 1/28; G 01 N 15/08; G 01 N 33/24 | | |
| II. FIELDS SEARCHED | | |
| Minimum Documentation Searched ⁷ | | |
| Classification System | Classification Symbols | |
| IPC ⁴ | G 01 N 1/00; G 01 N 15/00; G 01 N 33/00 | |
| Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched * | | |
| | | |
| III. DOCUMENTS CONSIDERED TO BE RELEVANT * | | |
| Category * | Citation of Document, ¹¹ with Indication, where appropriate, of the relevant passages ¹² | Relevant to Claim No. ¹³ |
| A | US, A, 2617719 (C.R. STEWART) 11 November 1952, see column 5, lines 63-72 | 1 |
| A | US, A, 2995027 (G.G. BERNARD et al.) 8 August 1961, see column 3, lines 32-40 | 1 |
| A | US, A, 2842958 (A.T. SAYRE JR et al.) 15 July 1958, see column 2, lines 24-63; figure 1 | 8 |
| A | US, A, 3839899 (J.M. McMILLEN) 8 October 1974, see column 2, line 50 - column 3, line 52; figures 1,2 | 8 |
| A | US, A, 4487056 (B.F. WILEY) 11 December 1984, see column 8, lines 1-25; figure 2 | 8 |
| ----- | | |
| <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: ¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p> </div> </div> | | |
| IV. CERTIFICATION | | |
| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report | |
| 6th May 1987 | 22 MAY 1987 | |
| International Searching Authority | Signature of Authorized Officer | |
| EUROPEAN PATENT OFFICE | M. VAN MOL  | |

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON

INTERNATIONAL APPLICATION NO. PCT/GB 87/00084 (SA 16013)

This Annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 11/05/87

The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|--|---------------------|----------------------------|---------------------|
| US-A- 2617719 | | None | |
| US-A- 2995027 | | None | |
| US-A- 2842958 | | None | |
| US-A- 3839899 | 08/10/74 | None | |
| US-A- 4487056 | 11/12/84 | US-A- 4552011 | 12/11/85 |

For more details about this annex :
see Official Journal of the European Patent Office, No. 12/82